Introduction

A procedure for unfilling used lithium lenses to has been described in Pbar Note 664 [1]. To date, the procedure has been used to disassemble lenses 20, 21, 17, 18, and 16. As a result of this work, some parts of the original procedure were found to be time consuming and ineffective. Modifications to the original procedure have been made to streamline the process and are discussed in this note. The revised procedure is included in this note.

Discussion

In the procedure presented in pbar NOTE 664 [1], lithium removal from the lens body was accomplished in two steps or phases. In Phase 1, vent/drain line plumbing tubing and valves were attached to the lens body fill ports. Heat was applied to the lithium lens body and the vent/drain line plumbing in order to raise the lithium temperature well above its melting point. With melted lithium in the lens body, argon gas pressure (as high as 40 psig) was applied to one side fill port which was intended to force lithium metal from the other fill port where it would be captured in an oil bath. The intended endpoint of Phase 1 was that most of the lithium (>90%) was removed and there would remain a flow path for argon gas from one fill port, across the lens body, and out the other fill port. At that point, the lens body was allowed to cool to room temperature. In Phase 2, a tubing pump and Bell jar were connected to the lens body fill ports. Water was pumped through the lens body where it would react with lithium and form lithium hydroxide and hydrogen gas. The resulting hydrogen gas was to be collected in a Bell jar for subsequent analysis in Phase 3. Eventually, all lithium metal would react with water leaving lithium hydroxide solution and otherwise clean steel and titanium parts. The lens body was drained and flushed with DI water to remove lithium hydroxide solution. The lens body, with all lithium removed could then simply be disassembled for examination.

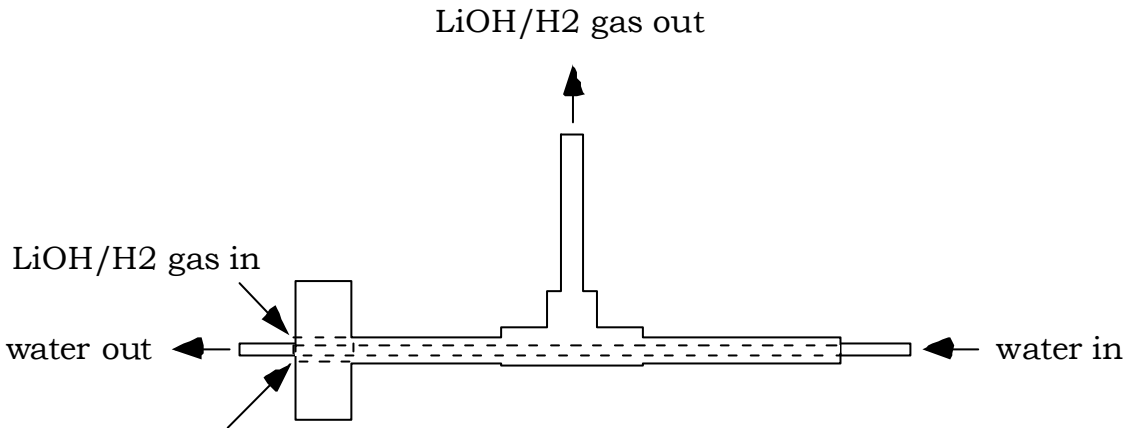
Attempts to removed lithium using the Phase 1 technique were only moderately successful. The main problem with Phase 1 was that the vent/drain line plumbing often became plugged with non-

lithium metal impurities. The nature and source of these impurities were discovered as experience with the unfilling process accumulated. Potential sources of impurities include lithium compounds formed due to lithium/water reaction after a septum failure, burned/oxidized titanium at the site of the septum failure, and mica from the current divider structure. Once a drain/vent line became plugged, it was necessary to cool down and disassembly the drain/vent line plumbing to remove the plug. It is possible that higher gas or oil pressure could have been used successfully to force out the melted lithium. However, it might have been relatively complicated and hazardous to do so in a controlled atmosphere glove box.

In reference 1, the premise for completion of Phase 1 was that there had to be a continuous gas flow path across the lens body. When it became clear that this was not achievable (Lens 17 had a hard plug which could not be removed), a new method was conceived which permitted removal of lithium metal with water without requiring a continuous flow path across the lens fill ports.

A nested tube system (see Figure 1) was built in which water could be introduced into a fill port through an inner tube while allowing lithium hydroxide solution and hydrogen gas to exit through an outer tube at the same fill port. This system allowed lithium metal removal without having a continuous flow path across the lens body. The resulting hydrogen gas was collected in the Bell jar as originally conceived and the lenses could be unfilled in spite of the blockage. This method was used for lenses 17 and 18 after unsuccessful attempts to establish a flow path with Phase 1. The method was used to unfill Lens 16 completely without any attempt to use Phase 1. This nested tube system has obviated the need for Phase 1 entirely.

The procedure for unfilling solid lenses has been revised. The number of phases is reduced from 4 to 3.



The phases are referred to as alpha characters instead of numeric ones to avoid confusion between versions. So, the Phases are labeled A, B, and C instead of 1, 2, 3, and 4. The Phase A, B, and C procedures follow immediately.

References

1. A Practical Method for Unfilling a Solid Lens, pbar Note 664 , A. F. Leveling, 6/4/01

Phase A Procedure

Refer to Attachment 2 for Phase A procedure setup.

NOTE: If a fire occurs during the unfilling process, evacuate the AP0 service building and call the fire department.

1. Install Phase A equipment in glove box including water tank and Bell jar, tubing pump, lens with nested tube fixtures, and necessary tubing and valves.

2. Install Phase B equipment on a table adjacent to the glove box and connect hydrogen line from glove box to the hydrogen inlet on the Bunsen burner.

3. Reestablish Argon atmosphere in glove box.

4. Connect tubing pump and hydrogen gas collection system to the lens body.

   NOTE: The water system is vented into a Bell jar so that hydrogen gas pressure will not build up and blow water line connections from the circulating water system.

   NOTE: Approximately 410 kcal heat is produced when 100 cc of lithium metal is reacted in water. Assuming no heat losses from the water system, the temperature of 7 gallons of water will rise approximately 16 ºC.

   Caution: Assuming the lens body contains 100 cc of lithium metal (see attachment 1), 80 to 90 liters of H2 gas with be produced due to the water reaction with lithium metal. Since the Bell jar capacity is approximately 18 liters, Phase A of the process must be discontinued periodically while the hydrogen gas is removed from the Bell jar in the Phase B process.

   Caution: In steps 4 through 12, hydrogen gas will be produced and collected in the Bell Jar. If at any time, the production of gas becomes excessive, discontinue

operation of the tubing pump and allow the hydrogen gas production to subside before proceeding.

NOTE: The water pH will increase from 7 to 14 as potentially radioactive lithium metal reacts with circulating water. Use appropriate precautions when handling caustic, potentially radioactive solutions.

NOTE: Initially, the hydrogen gas production rate will not be significant since the surface area exposed to water will be quite small. The surface area will increase as lithium metal is reacted away, and the hydrogen gas production rate will increase according. Care must be taken to control the hydrogen production rate by limiting the introduction of water to the lens body.

CAUTION: the return side of each nested tube arrangement must remain open once water has been introduced to its respective side so that hydrogen gas may be freely vented to the Bell jar.

5. Position the lens body with the fill ports up.

6. Open the water supply valve to one fill port through the nested tube water supply line.

7. Energize the tubing pump at minimum speed to just establish water flow.

8. Monitor water flow from the tank to the fill port and out of the return line.

9. Note release of hydrogen gas from lens body by formation of gas bubbles in Bell jar.

NOTE: Pumping speed may be increased as the water volume in the lens body increases in order to promote mixing.

10. Monitor the gas volume in the Bell jar. As the gas bubble approaches 10 inches in height, stop the pump. Invert the lens body so that the fill ports are oriented in the down position.

Reverse the pump direction and energize the pump until water removal from the lens body ceases.

11. Shut the water supply valve to the nested tube fixture.

12. Proceed to Phase B procedure to consume hydrogen gas.

13. When hydrogen gas has been removed from the Bell jar, repeat steps 5 – 12.

NOTE: After three to four burn cycles, the lithium should be reacted at least half way through the lens body. Water exchange limitations will slow down exchange of fresh water on the lithium metal surface with a corresponding reduction in the gas production rate. Water should be introduced in the second fill port.

NOTE: After five to six burn cycles, water flow should be possible from one port through the lens body and out the second fill port. When evidence of gas flow through both return lines appears, proceed to step 14.

14. Stop the tubing pump.

15. Ensure return valves are open for each fill port.

16. Shut the water supply valve to one fill port.

17. Turn on the tubing pump at very slow speed.

CAUTION: When the return tubing is pinched, pressure may build up if the flow path is not open through the lens body between the fill ports. If the path is open flow will be observed through the opposite side return path.

18. Momentarily pinch the return tubing from the side being supplied with water by the tubing pump and observe whether flow is established through the opposite side return path.

19. If flow is observed in the previous step, shut the return valve on the side being supplied with water and observe the return flow is established in the opposite side.

20. If flow is not established, keep both supply and return valves open until evidence of flow is seen at the opposite side fill port return line.

21. After flow is established through the lens body and all evidence of gas production has ceased, proceed to Phase B to complete gas consumption.

22. Slowly pump 1 liter of clean DI water through the lens body and septum cooling water circuit and allow to discharge into Bell jar reservoir or an alternate container if necessary.

23. Stop tubing pump.

24. Start the tubing pump and collect a 50 milliliter sample of water by momentarily energizing tubing pump and collecting the effluent in a beaker.

25. Check the effluent pH and note.

26. If the pH is less than 9, lithium metal removal is complete. Proceed to step 29.

27. If the pH exceeds 9, the lens body may require a second flush (step 22). Alternately, if there is evidence of gas production, the lens may require additional cleaning time (step 13).

28. Disconnect pump suction from Bell jar and direct discharge to Bell jar.

29. Energize pump to push remaining water out of lens body with Argon.

30. Stop pump.

31. Close valves on Bell Jar vent line to maintain gas volume.

32. Disconnect discharge line from lens body to Bell Jar vent line.

33. Reverse pump direction and direct pump suction to Bell jar.

34. Energize pump to remove water from low point.

35. Stop pump

36. Establish an argon gas purge through the lens body and septum cooling water circuit to dry out the lens body assembly.

37. Remove lens body from glovebox.

Phase B Procedure

Refer to Attachment 3 for Phase B procedure.

NOTE: If a fire occurs during the unfilling process, evacuate the AP0 service building and call the fire department.

1. Ensure valves are closed on Bell jar tubing to preserve hydrogen gas collected in the Bell jar.

2. Establish cooling water flow to Graham condenser.

3. Establish air flow through chimney by energizing water filtration pump.

4. Light propane fueled Bunsen burner and establish a flame height of 1.5 to 2 inches by adjusting fuel inlet needle valve and air mixing sleeve.

   CAUTION: the glass funnel and chimney will become very hot due to burner operation. Do not handle the glass funnel or chimney until after the burner gas flow has been stopped and these components have been permitted to cool.

5. Allow burner to operate for 5 minutes and note collection of water in Erlenmeyer flask.

6. Crack open Bell jar vent valve to slowly introduce hydrogen gas into Bunsen burner.

7. Continue introduction of hydrogen gas into Bunsen burner flame until all gas in the Bell jar is consumed and water just reaches the vent line opening in the Bell jar.

   NOTE: Add water to water tank if necessary to force hydrogen gas from Bell jar into burner flame.

8. Close Bell jar vent valve.

9. Stop gas flow to Bunsen burner and ensure flame is extinguished.

10. Return to Step 13 of Phase B procedure if additional lithium metal is present in the lens body.

11. Allow chimney and funnel to cool.

12. Collect water in Erlenmeyer flask for further analysis.

13. If tank water is required to be analyzed for tritium, proceed to Phase C.

Phase C Procedure

Lithium Hydroxide Solution Neutralization Procedure

Required MSDS sheets:

- Hydrochloric acid
- Phenolphthalein

Equipment:

- Scale, 50 lb capacity or sufficient capacity for weighing solution containers
- 250 cc Erlenmeyer flask
- 100 cc microburrette with aspirator bulb
- 100 cc pipette with aspirator bulb
- small glass funnel
- disposable pipette

Materials:

- Hydrochloric acid
- 0-14 pH paper
- Phenolphthalein color indicator (pH=8)

The empty weight of the container holding the LiOH solution (hereafter referred to as container) must be determined. An empty container of the same type (e.g., bucket, pail, beaker, carboy, etc.) may be used for this determination. Sufficient free volume in the container must be available for additions of hydrochloric acid so that the solution may be neutralized.

1. Pump LiOH solution from Bell jar reservoir to 5 gallon plastic. Carboys.

2. Neutralize LiOH solution with HCl to pH range of 5 to 9 as follows:

CAUTION: Hydrochloric acid may cause serious injury or death if mishandled. Review MSDS for proper handling before performing this procedure. Take proper precautions before using any chemicals required for this procedure.

1. Determine the desired ending pH range of the LiOH solution and record it. _____
2. Determine the empty weight of the container and record the weight in pounds. _____ lbs (A)
3. Weight the container to be neutralized and record the weight in pounds. _____ lbs (B)
4. Fill microburrette with HCL solution to be used in neutralization. Use the glass funnel to control filling.
5. Note beginning level of HCl in microburrette. _____ ml (C)
6. Transfer 100 cc of solution to be neutralized to Erlenmeyer flask.
7. Add one or two drops of color indicator to the solution and mix. Color should turn pink.

NOTE: If the color does not turn pink, the solution pH is already less than 8 and this procedure should be discontinued.

8. Slowly begin addition of HCl by microburrette drop-wise. The stopcock on the burrette can be adjusted to permit a few drops per second. Mix solution as HCl is added.

9. When color changes from pink to colorless, close microburrette stopcock.
10. Continue mixing. If solution remains clear, go to step 13.
11. If solution returns to pink, open stopcock and slowly add HCl about 1 drop per second.
12. Repeat steps 10 through 12 until solution remains colorless.
13. Record the ending burette level. _____ ml (D)
14. Return neutralized sample to the LiOH solution container
15. Calculate the quantity of HCl solution to add to the container as follows

$$\frac{[B-A]*[D-C]}{0.22} = mlHCl$$

and record the volume here:_____ ml HCl

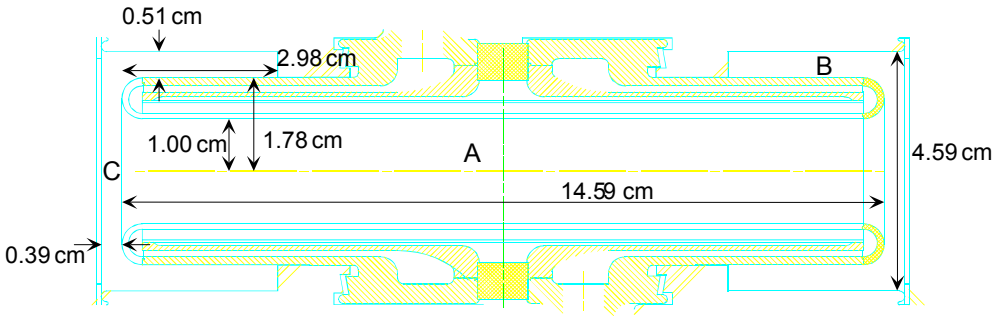
16. Pour the volume of HCl calculated in step 15 into the container and mix thoroughly.
17. Insert a pH paper strip into the container briefly and remove. Compare the color pattern on the strip to the one on the pH paper case and note the pH. _____

The pH should be about 8 plus or minus one pH unit. Other color indicators are available for pH endpoints and may be substituted. If the ending pH is higher than desired range noted in step 1, repeat steps 2 through 17. If the pH is lower than the desired range, the pH may be adjusted upward by the addition of a basic solution such as sodium hydroxide. If an adjustment is necessary, the weights noted in steps 1 and/or 2 may not be have been accurately determined.

3. Collect samples of neutralized solution for analysis of tritium and accelerator produced isotopes.

NOTE: Water used to dissolve lithium may need to be disposed of as radioactive waste depending up sample collected in Phase A.

NOTE: Other lithium compounds may be present and fixed at the site of the breach. Removal of such materials should be made in a passive manner so that septum surfaces are not altered by this removal.



NOTE: Assume fill ports arenegligible

Volume calculation

Volume A = $\pi r^2 l$
r = 1 cm
l = 14.59 cm

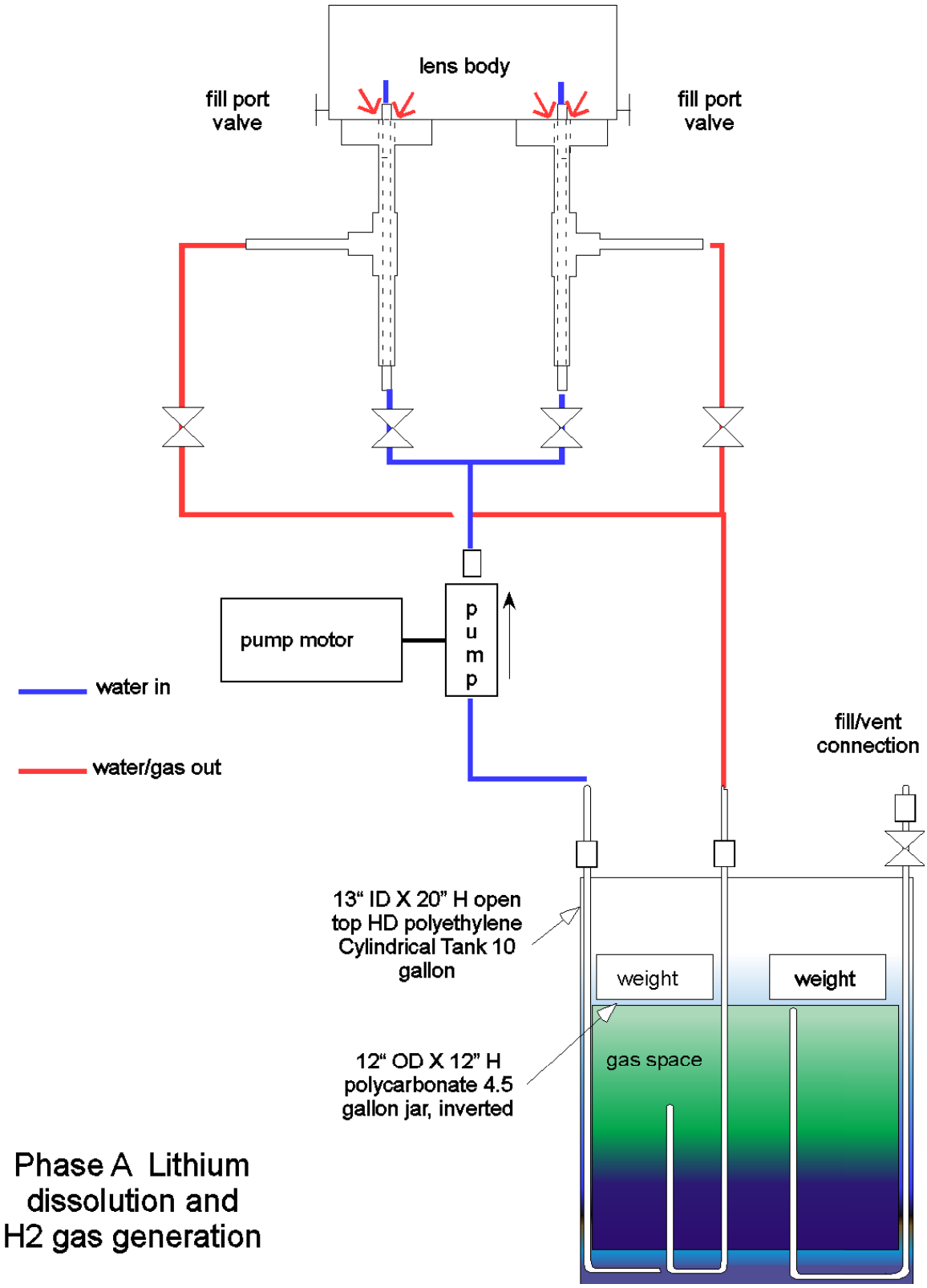
Volume B = $2\pi(d/2^2 - r^2)l$
d = 4.59 cm
r = 1.78 cm
l = 2.98 cm

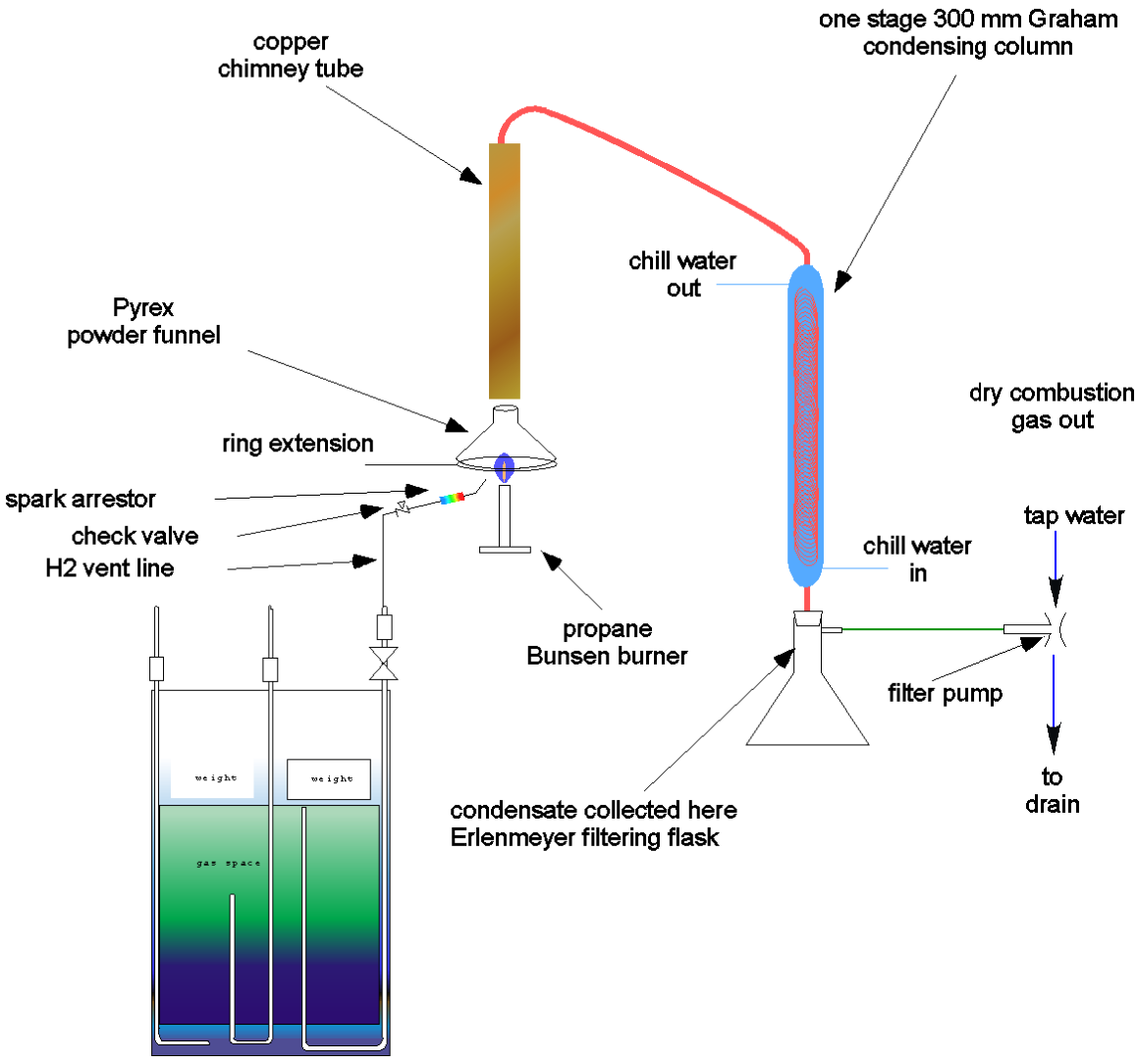
Volume C= $2\pi(d/2)^2 l$
d= 4.59 cm
l = 0.39 cm

Total Volume = A + B + C = 98cc

Attachment 1

prepared by T. L eveling
6/26/00


lens body
fill port valve
fill port valve
pump motor
p u m p
water in
water/gas out
fill/vent connection
13“ ID X 20” H open top HD polyethylene Cylindrical Tank 10 gallon
weight
weight
gas space
12“ OD X 12” H polycarbonate 4.5 gallon jar, inverted
Phase A Lithium dissolution and H2 gas generation



Collection Lens Body Unfilling Plan
PHASE B